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(Triannual Report)

DEVELOPMENT OF SPACE-STABLE THERMAL-CONTROL COATINGS

National Aeronautics & Space Administration
George C. Marshall Space Flight Center
Huntsville, Alabama

IIT RESEARCH INSTITUTE

Report No. IITRI-U6002-51
(Triannual Report)

DEVELOPMENT OF SPACE-STABLE THERMAL-CONTROL COATINGS

Contract No. NAS8-5379
IITRI Project U6002

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Prepared by
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Submitted by
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Huntsville, Alabama

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FOREWARD

This is Report No. IITRI-U6002-51 (Triannual Report) of IITRI Project U6002, Contract No. NAS8-5379, entitled "Investigation of Environmental Effects on Coatings for Thermal Control of Large Space Vehicles." This report covers the period from October 20, 1966 through February 20, 1967. Previous Triannual Reports were issued on October 25, 1963; March 5, 1964; July 20, 1964; December 21, 1964; February 23, 1965; July 20, 1965; November 9, 1965; February 21, 1966; July 11, 1966, and November 30, 1966.

Major contributors to the program during this period include Gene A. Zerlaut, project leader and, alphabetically; William C. Courtney, consultation on vacuum problems; George Kimura, vacuum technology and space simulation tests; Wayne Ridenour, vacuum equipment design; and Samuel Shelfo, reflectance measurements and space simulation tests. Dr. T. H. Meltzer, Manager of Polymer Research, provided administrative supervision. The work reported herein was performed under the technical direction of the Research Projects Laboratory of the George C. Marshall Space Flight Center with Daniel W. Gates acting as Project Manager.

Prior to March 15, 1966, this contract was funded under Codes 124-09-05-00-14, 933-50-01-00-00, and 908-20-02-01-47.

Respectfully submitted,
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GAZ/mc

DEVELOPMENT OF SPACE-STABLE
THERMAL-CONTROL COATINGS

I. INTRODUCTION

The general requirement under this contract is for the development of thermal-control surface coatings which possess very low but stable ratios of solar absorptance (α_2) to infrared emittance ($\mathcal{E}_h$). The program has been divided historically into three major phases; (1) inorganic technology, (2) silicone photolysis and silicone paint investigations, and (3) general coatings investigations.

The relative emphasis placed upon each of these three major tasks has varied during the course of the program in accordance with the urgency of the various problems elucidated by our investigations, as well as the availability of both funds and personnel. For example, the necessity for performing in situ reflectance measurements was emphasized by the results of both flight (ref. 1,2) and laboratory (ref. 3,4) experiments.

The design of an in situ facility was therefore initiated on this project early in 1966. The facility is described in detail in the last Triannual Report (IITRI-U6002-47) and in a paper (ref. 5) scheduled for presentation at the forthcoming AIAA Thermophysics Specialists Conference in New Orleans (April 17-19, 1967).

The work performed during this report period and discussed herein consists almost exclusively of final calibration and design-modification studies on the in situ facility described above, the IRIF (In Situ Reflectometer Irradiation Facility). Four successful space simulation tests have been completed to date and are summarized with data in the following paragraphs.

II. THE IRIF

A. Design Modifications

1. General

A considerably more stable optical interchange was constructed. This involved the removal of the cover and the standard integrating sphere from the DK-2A's reflectometer attachment. The cover was replaced with a similar plate from which two front surface mirrors were rigidly suspended -- one for the specimen beam and the other for the reference beam. The beams are directed upwards into the integrating sphere through the vacuum manifold; the mirrors are located in the space previously occupied by the DK-2A sphere. The construction of the stronger interchange have insured easier mating of the DK-2A and the IRIF and have thus far always provided positive optical alignment.

The two manipulator arms (A and B, Figure 27, Report IITRI-U6002-47) were refabricated from larger stock and were more securely attached to the atmosphere-side of the bellows. The manipulator arms that are attached to the vacuum side of these same bellows were spot welded.

The twelve hold-down pins that radiate from the spring-loaded shaft and hold the samples (see Figures 24 and 25, Report IITRI-U6002-47) were more sharply beveled in order to facilitate the easy attachment and removal of the substrates. The pin that is located in the center of the sample boat assembly was beveled for the same reason.

2. Detector Switch Construction

The DK-2A reflectance attachment is provided with two interchangeable detector heads -- an 1P-28 photomultiplier detector and a 10mm x 20mm lead sulfide cell.

Each detector assembly possessed a multiple-pin male connector.

The multiple position switch on the photomultiplier was redesigned as shown in Figure 1. The switch was adjusted to four positions; OFF, IR, UV-1X and UV-20X. The new switch assembly not only saves time in performing the measurements but, more important, saves wear on the multiple-pin connector assembly.

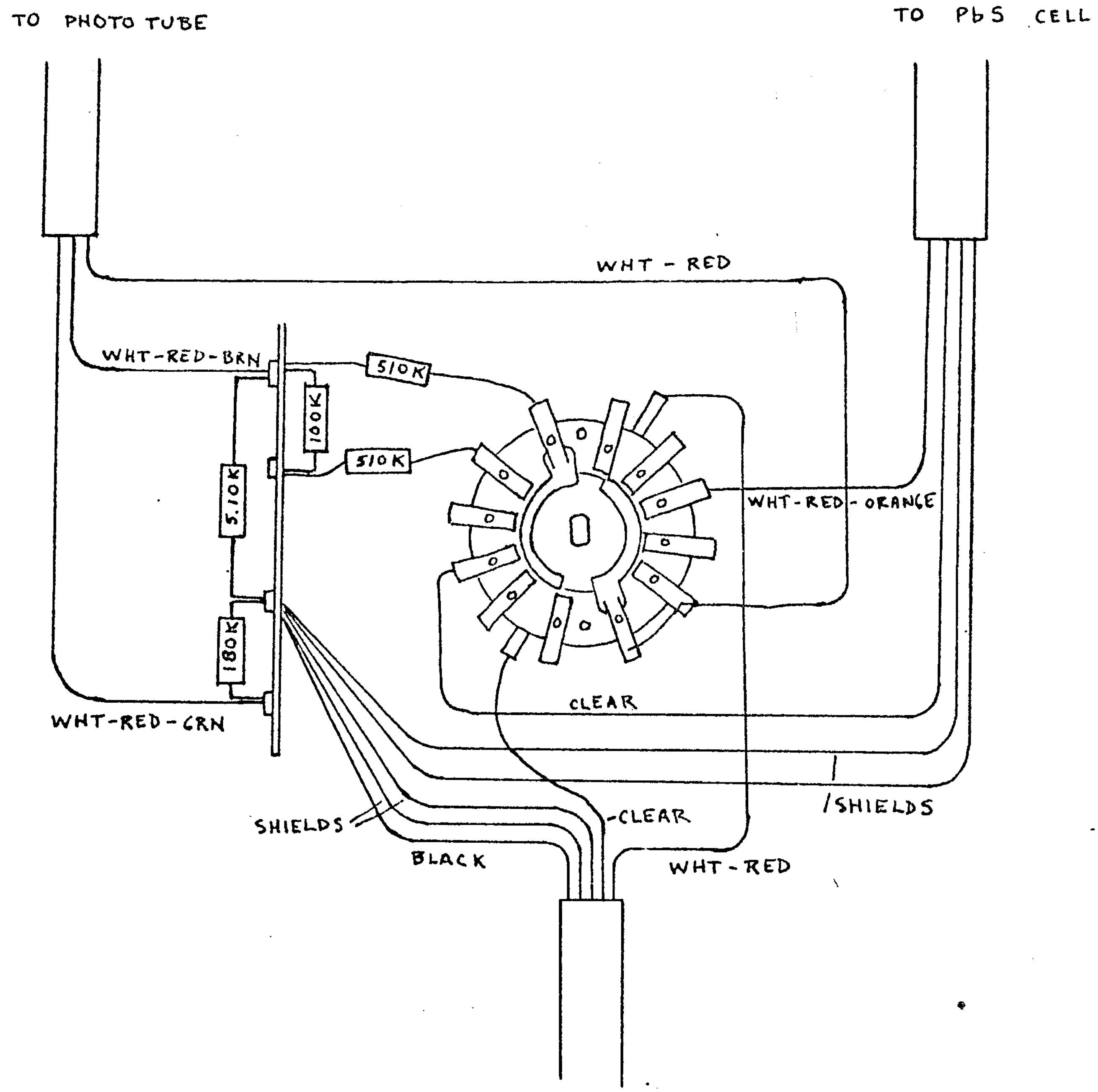
B. Functional Problems

1. Sphere-Detector Geometry

The reflectance of a polished aluminum sample substrate (blank) is shown in Figure 2. The discrepancy between the photomultiplier tube and lead sulfide cell operation indicates a strong sensitivity to the specularity of samples. This sensitivity is attributed to the fact that the active surface in the photomultiplier tube is mounted behind a glass envelope and thus is not hemispherically irradiated. Every attempt was made to mount the glass envelope sufficiently into the sphere to ensure that the active surface of the tube was positioned in a plane tangent to the sphere. Obviously the internally mounted lead sulfide cell is hemispherically illuminated. Techniques such as lightly etching the glass envelope will be investigated to decrease the directionality of illumination received by the photomultiplier tube.

The disparity recorded in Figure 2 has only been observed with specular, metallic samples. The reflectance at 0.7 μ differed at most by 1% between the photomultiplier- and lead sulfide-detector modes for all other specimens.

The loss in ultraviolet reflectance of the aluminum with irradiation is attributed to organic contamination. This specimen was in the first test; we later observed that some organic photolyzed on the window. The system was subsequently cleaned, evacuated and a "blank" ultraviolet



TO AMPLIFIER

Figure 1

SCHEMATIC OF REDESIGNED DK-2A REFLECTOMETER
DETECTOR SWITCH

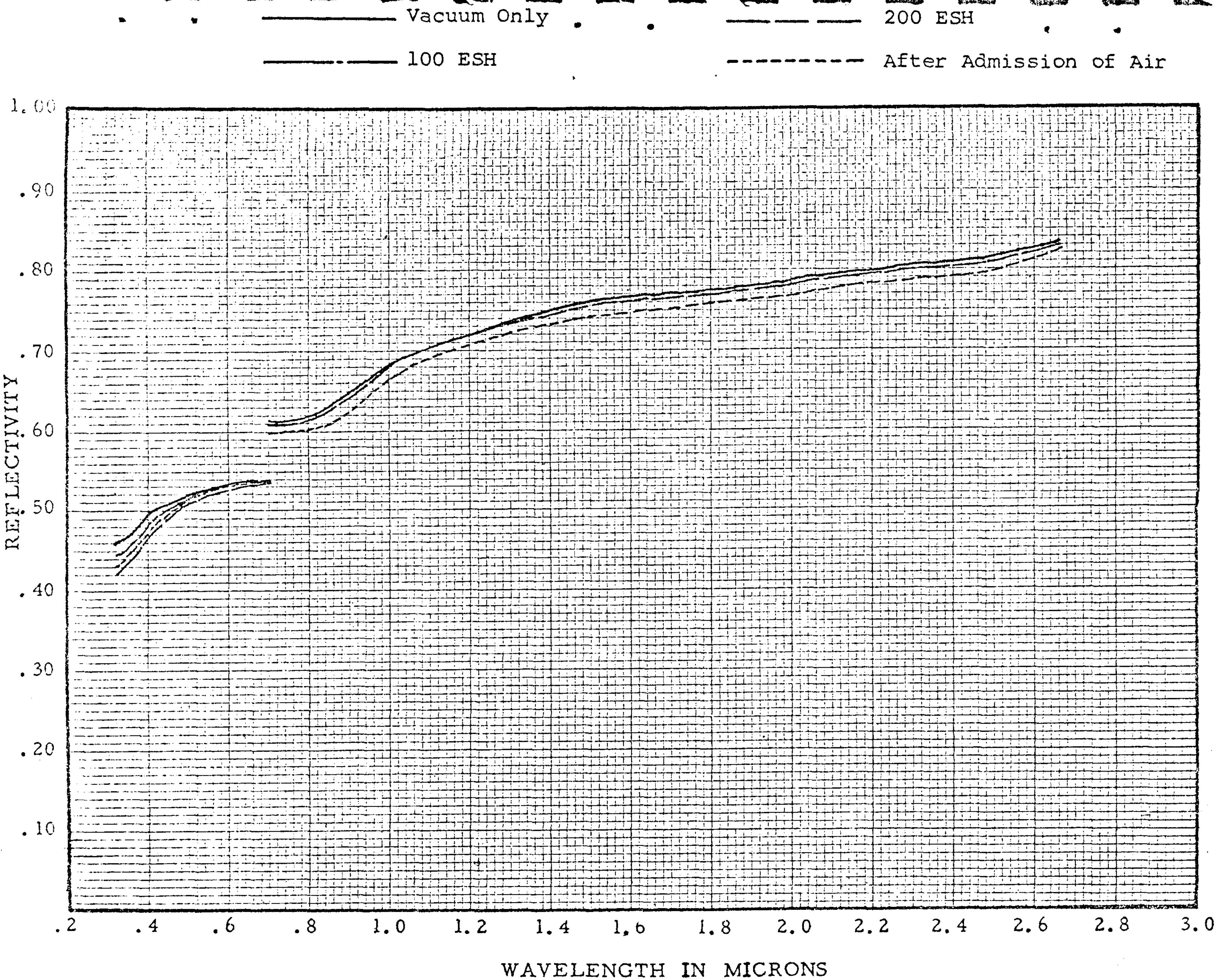


Figure 2: EFFECT OF 200 ESH UV IRRADIATION ON A SMOOTH ALUMINUM SUBSTRATE

test on aluminum performed. No organic deposits were thereafter noted.

2. Lead Sulfide Cell Degradation

A major concern during the design of the IRIF was the possibility that lead sulfide detectors would quickly and severely degrade in vacuum. Although we have examined only one detector assembly to date, a slow increase in the noise level and a decrease in the long wavelength slit control with increasing time in vacuum were observed. Because of the ratio recording operation of the Beckman, vacuum fatigue of the lead sulfide detectors is not expected to decrease the accuracy of the measurements, although excessive noise might. Any cell degradation could seriously affect the wavelength limits of the measurement, however.

The slit openings at 1.5-, 2.0- and 2.4-μ wavelengths are recorded as a function of time in vacuum in Figure 3. The increase in slits from 0.78 to 0.98 mm at 2.4-μ wavelength was manifested by a decrease from 2.65 and 2.5μ in the long wavelength limits of the measurement. This change required 600 hr of accumulated real-time evacuation. The noise level did not exceed the maximum permissible amplitude in this length of time. Note that 600 real-time hr represents 3500 equivalent sun-hours, if the irradiation intensity averages about 6X.

Since the 10- x 10-mm lead sulfide cells are inexpensive and require about an hour to replace, it was decided to leave the lead sulfide detectors on the inside of the integrating sphere -- at least until more data are collected.

C. IRIF Data

Four space simulation tests have been performed to date. The first two involved specimens prepared on both this program and the JPL program (Contract 951737, Project U6053).

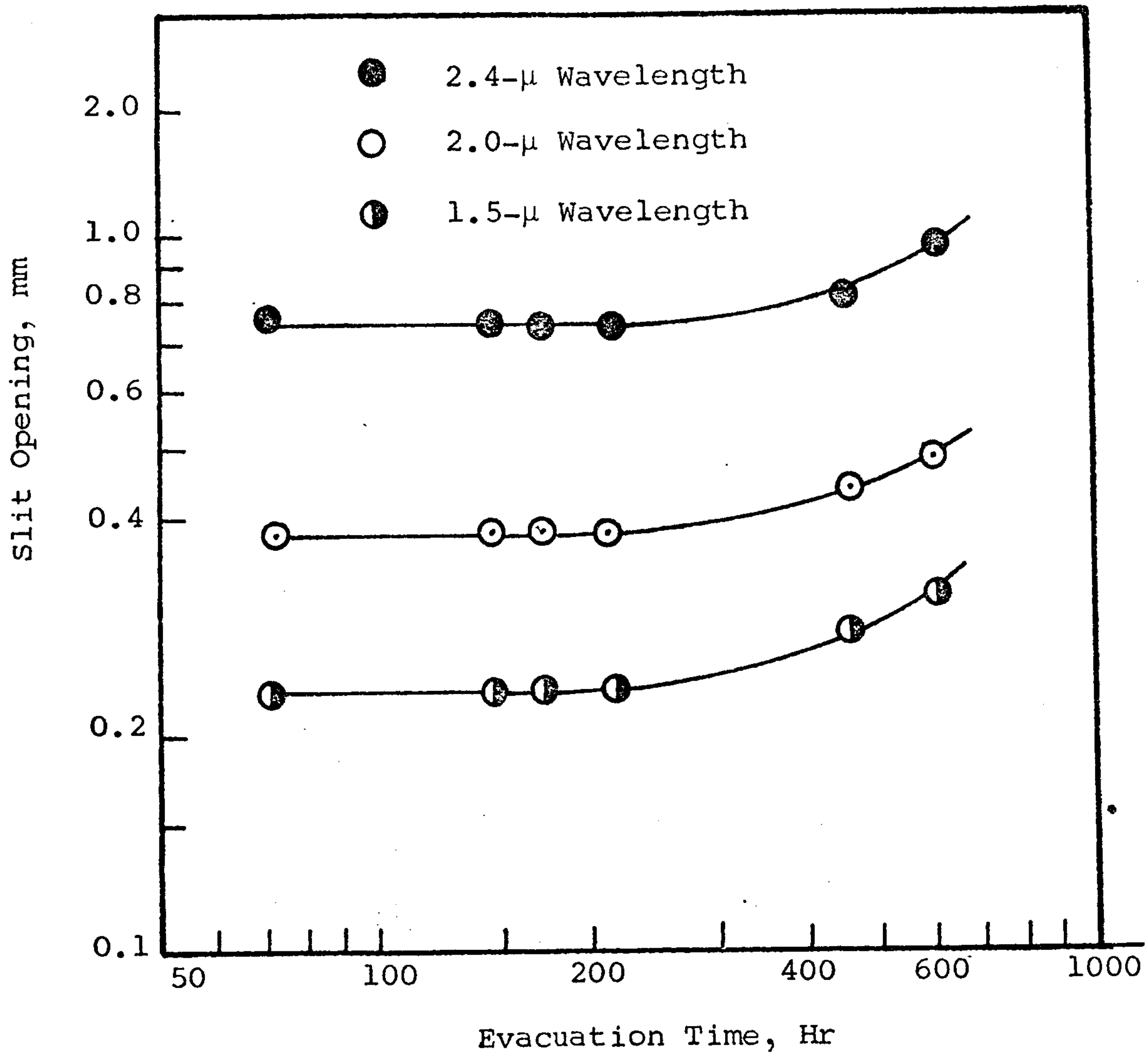


Figure 3 - EFFECT OF EVACUATION AT 10^{-7} TORR ON THE OUTPUT OF TWO SERIES-MOUNTED 10 mm X 10 mm LEAD SULFIDE CELLS

The third test was aborted due to failure of the AH-6 lamp assembly which resulted in the burner resting on the Suprasil window. The proximity of the burner to the window allowed ultraviolet to attack the Viton O-ring used to seal the window. This is the only non-metal seal used on the IRIF and is normally shielded from ultraviolet radiation. The chamber became contaminated during this test and had to be cleaned with a 5% solution of NaOH followed by ethyl alcohol. The facility was subsequently evacuated (Test 4) with aluminum blanks (as samples) and irradiated for 24 hours. No organic deposits were observed.

The first two tests were each for 200 ESH of ultraviolet irradiation; the irradiation pressure in each case was approximately 1×10^{-7} Torr. The only difference between the two tests was the air that was admitted at the completion of the test. Moist, ambient room air was admitted at the completion of Test 1 whereon compressed prepurified air was admitted at the completion of the second test. Reflectance values were taken after 100 ESH of irradiation in Test 1 but only at the completion of irradiation in Test 2.

The results of these two IRIF space-simulation tests are summarized by the data presented in Figures 4 through 10. Some general observations of these tests are:

(1) The reflectance of zinc oxide powders and zinc oxide paints increased after irradiation in the ultraviolet region below zinc oxide's absorption edge.

(2) Admission of air to the facility increased the reflectance in the visible region for most specimens, whereas, for specimens not exhibiting bleachable infrared degradation, the reflectance decreased at wavelengths beyond 0.8 to 0.9 microns.

(3) Admission of air also causes the reflectance of

zinc oxide specimens to decrease in the ultraviolet region below its absorption edge.

(4) Irradiation caused a loss of fine structure to specimens exhibiting structure in the infrared. Admission of air, however, increased the fine structure to a degree which usually surpassed that of the unirradiated specimen. See Figures 4, 6 and 7.

(5) Admission of moist air also resulted in the creation of a water band at approximately 1.95 microns for silicate-containing specimens. See Figure 7.

The spectra obtained for S-13 (Figure 4), SP500 zinc oxide powder (Figure 5) and S-13G (Figure 6) were essentially identical to those obtained in the in situ facility employed earlier (in which the reflectance of specimens was measured through a window).

The specimen of S-13G over S-13 exhibited an increase in reflectance in the visible in the first 100 ESH of irradiation. This specimen was prepared on another contract and is included here for purposes of illustration. The bleachable damage in the infrared was partially masked by the formation of the absorption band at about 1.95 microns on admission of moist air.

The zirconia powder (Figure 8) and the zinc titanate powder (Figures 9 and 10) specimens did not exhibit bleachable infrared degradation in the infrared. The damage exhibited by the zirconia was somewhat greater than is usually observed after 200 ESH when the postexposure measurements are performed in air. This is evidenced by the behavior of the specimen on admission of air; a 9% increase in reflectance was observed at 4000-A wavelength.

The most encouraging results of the second IRIF test was the stability exhibited by the two specimens of

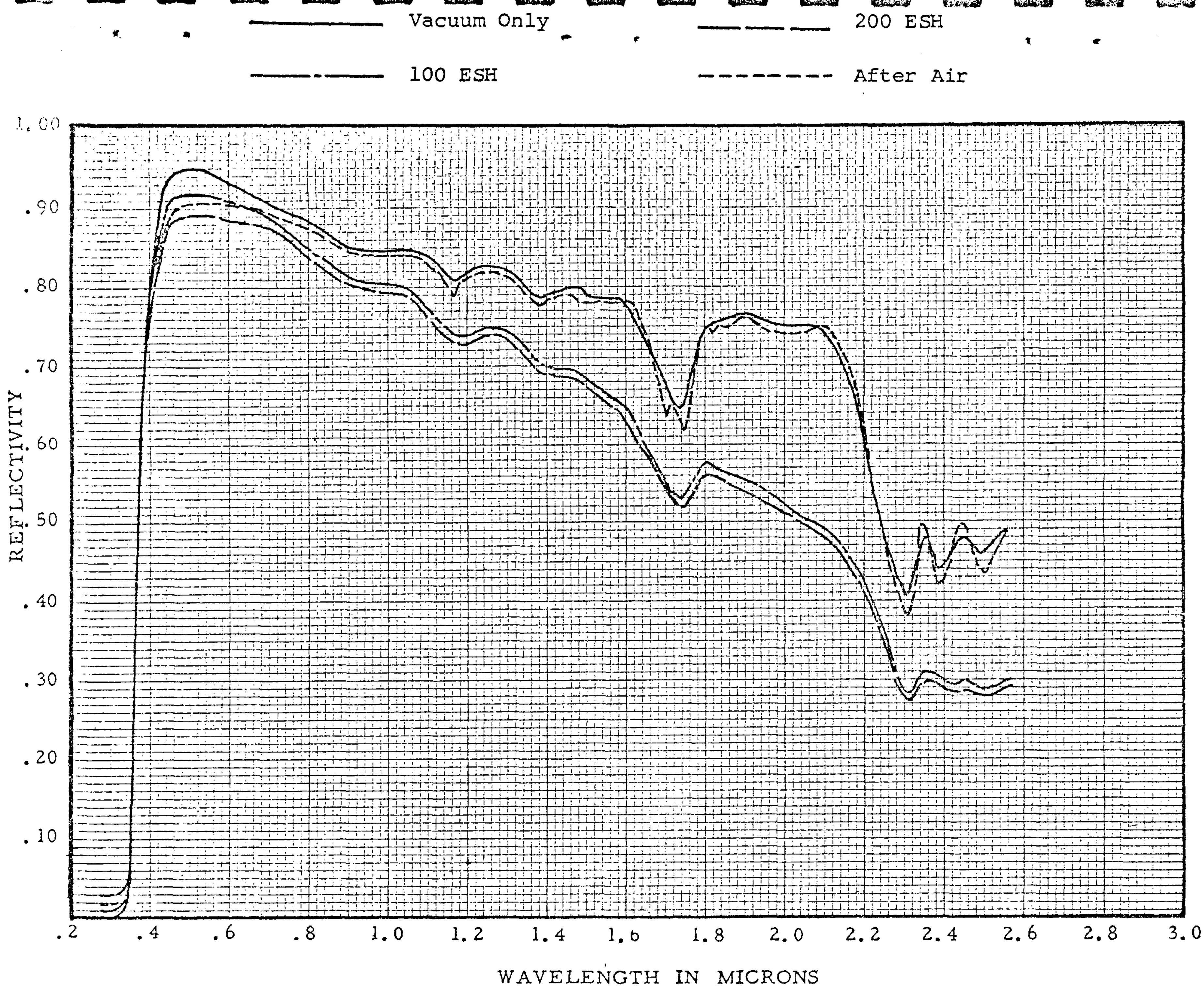


Figure 4: EFFECT OF 200 ESH UV IRRADIATION ON S-13 (IRIF TEST 1)

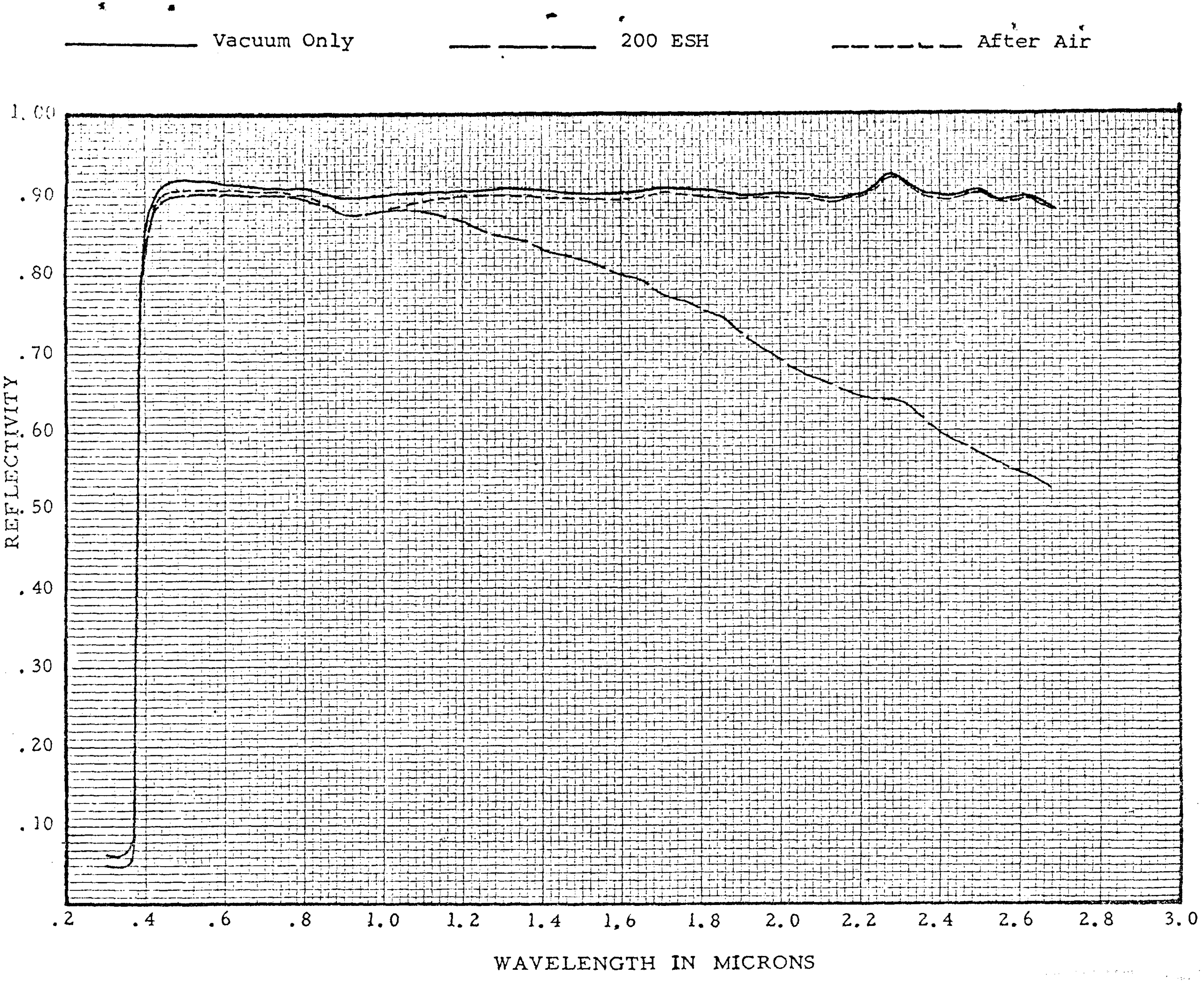


Figure 5: EFFECT OF 200 ESH UV IRRADIATION ON SP-500 ZnO (IRIF TEST 2)

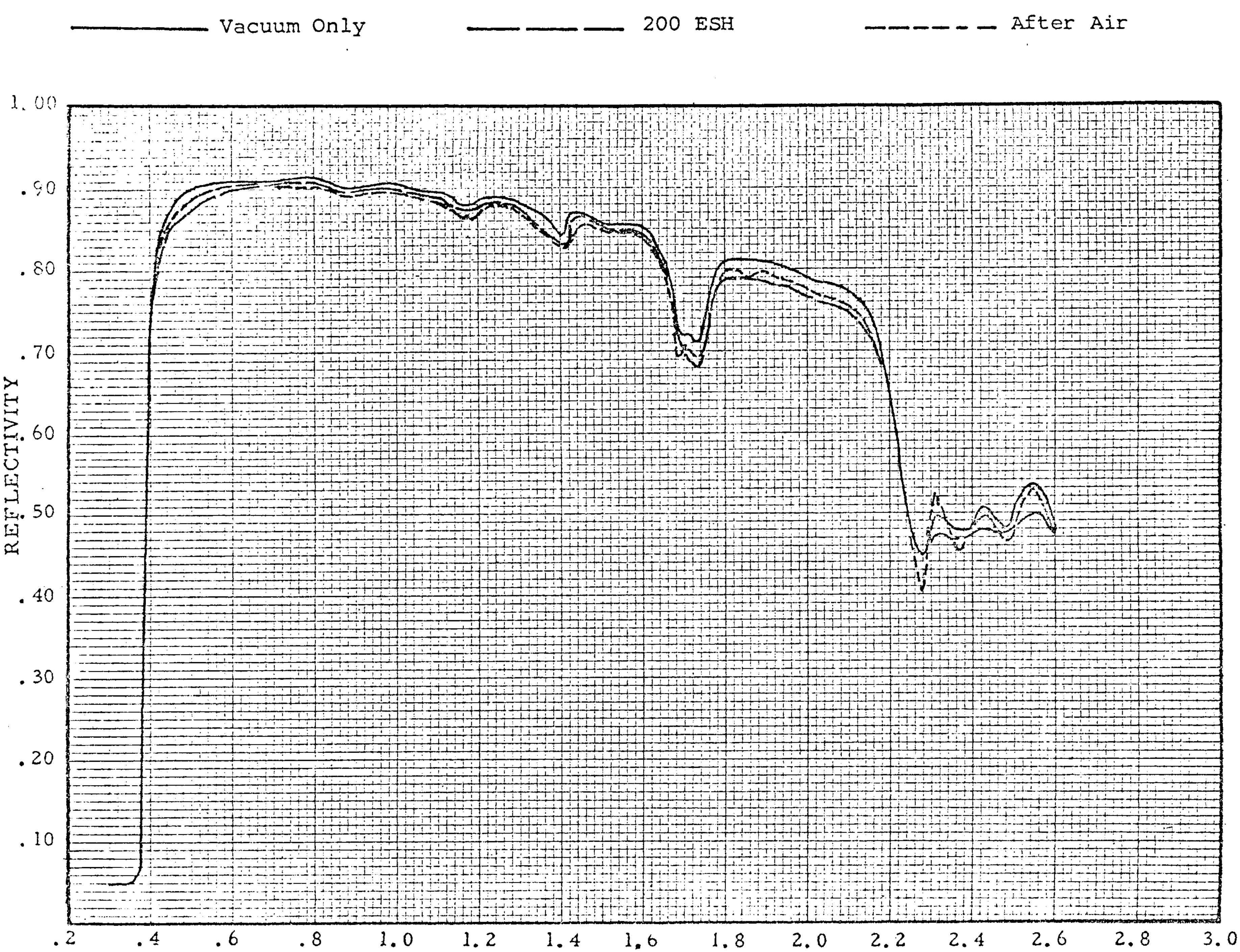


Figure 6: EFFECT OF 200 ESH UV IRRADIATION ON S-13G (IRIF TEST 2)

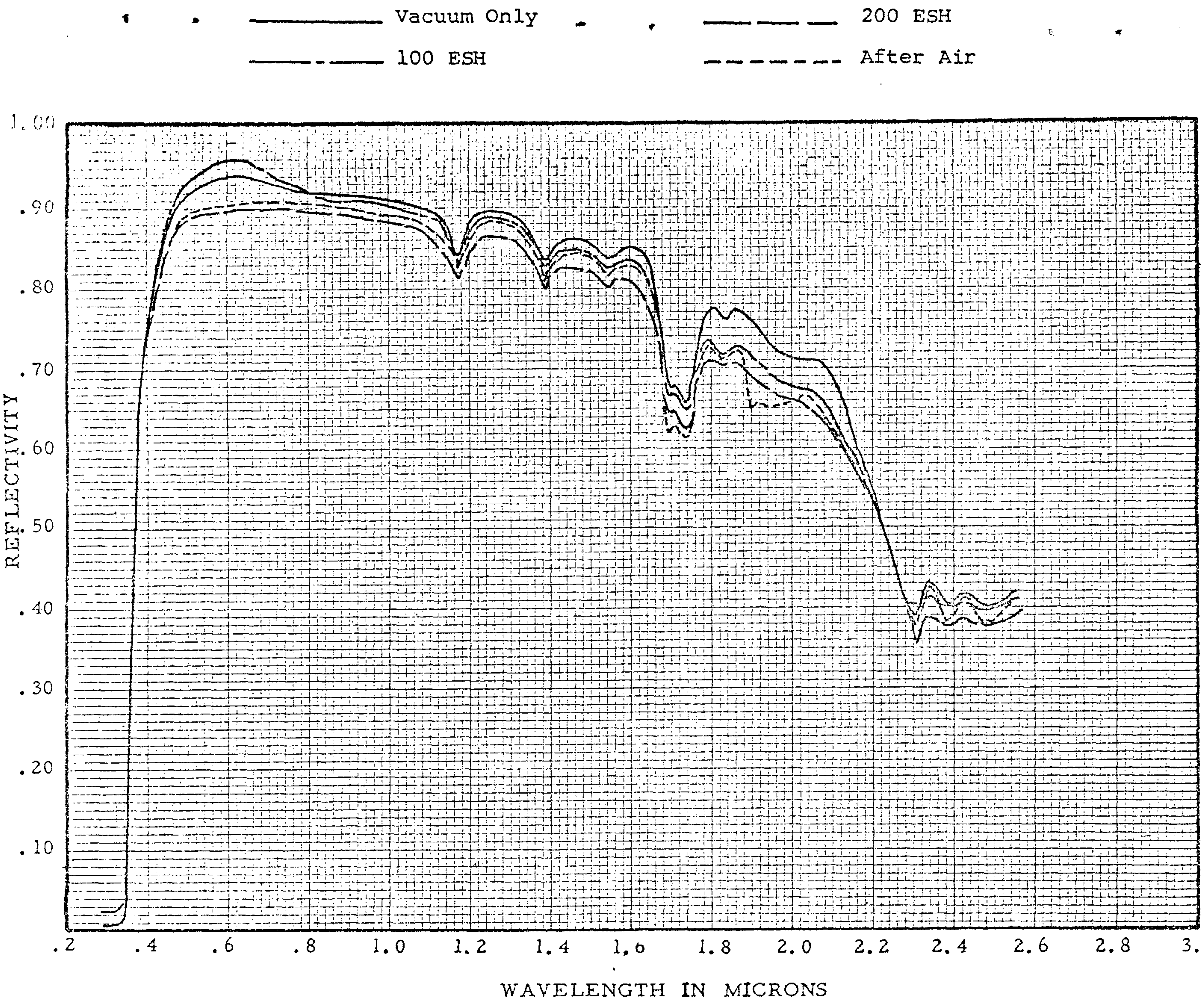


Figure 7: EFFECT OF 200 ESH UV IRRADIATION ON S-13G OVER S-13 (IRIF TEST 1)

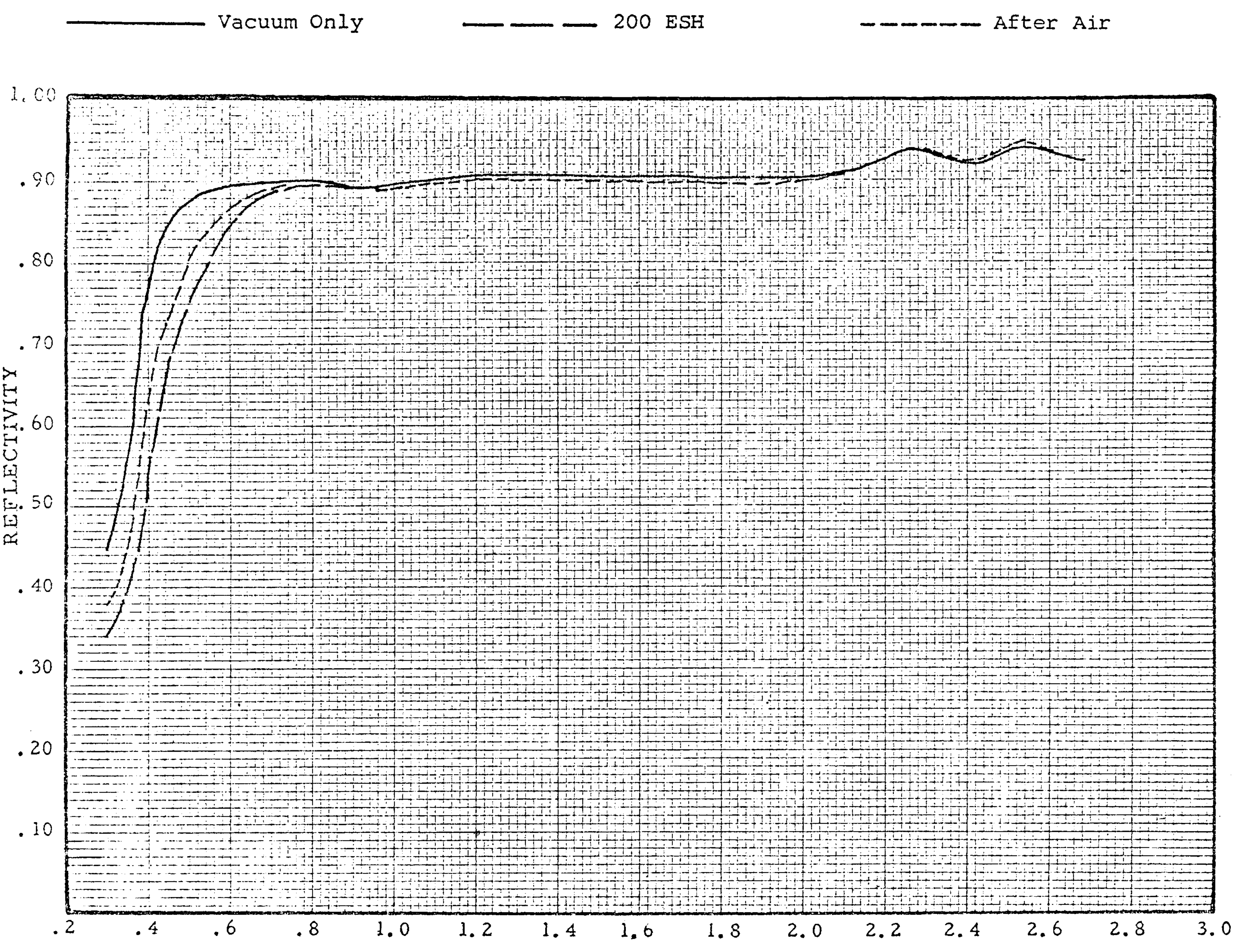


Figure 8: EFFECT OF 200 ESH UV IRRADIATION ON Z_rO_2 POWDER (IRIF TEST 2)

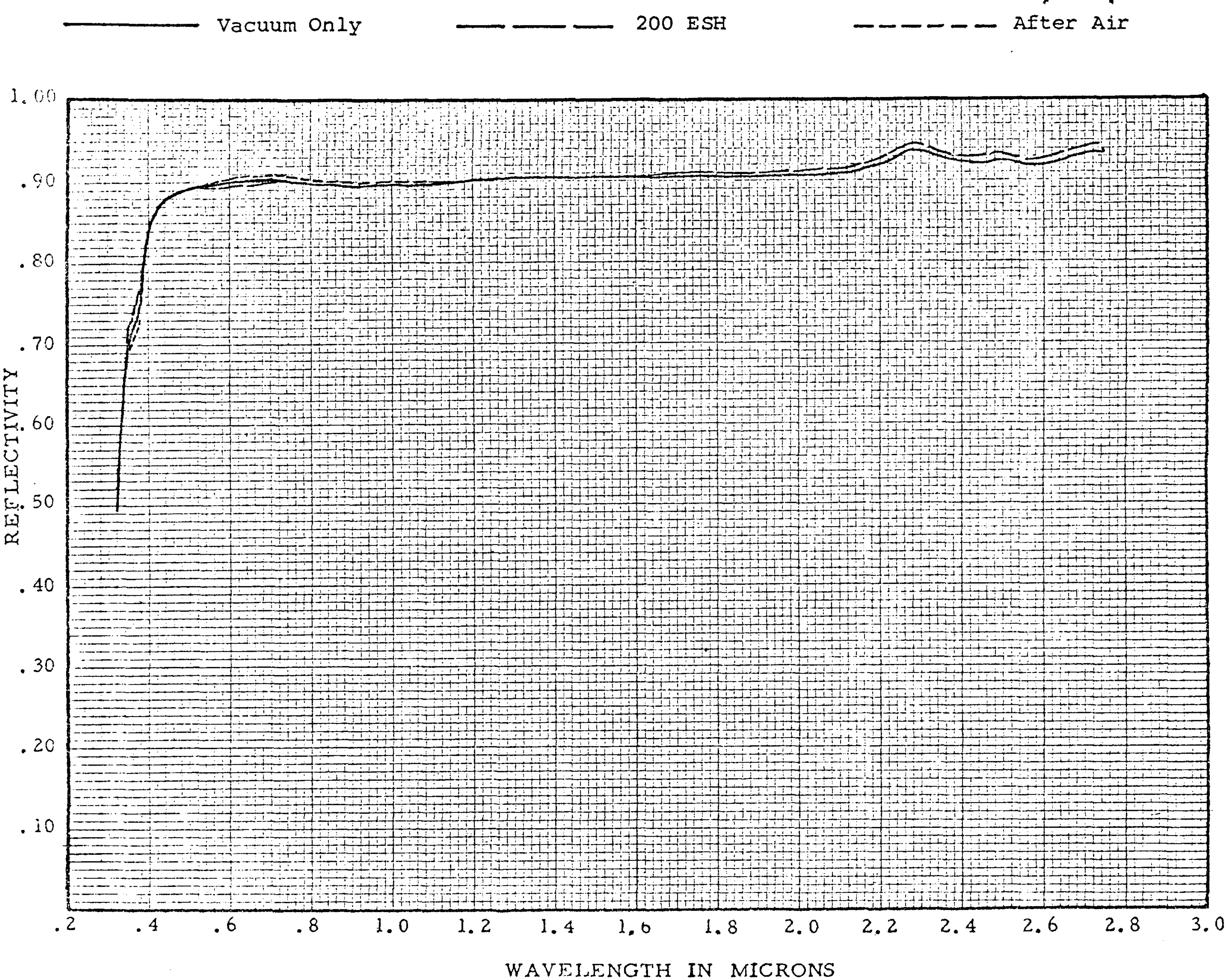


Figure 9: EFFECT OF 200 ESH OF UV IRRADIATION ON Zn_2TiO_4 POWDER (SAMPLE A IRIF TEST 2)

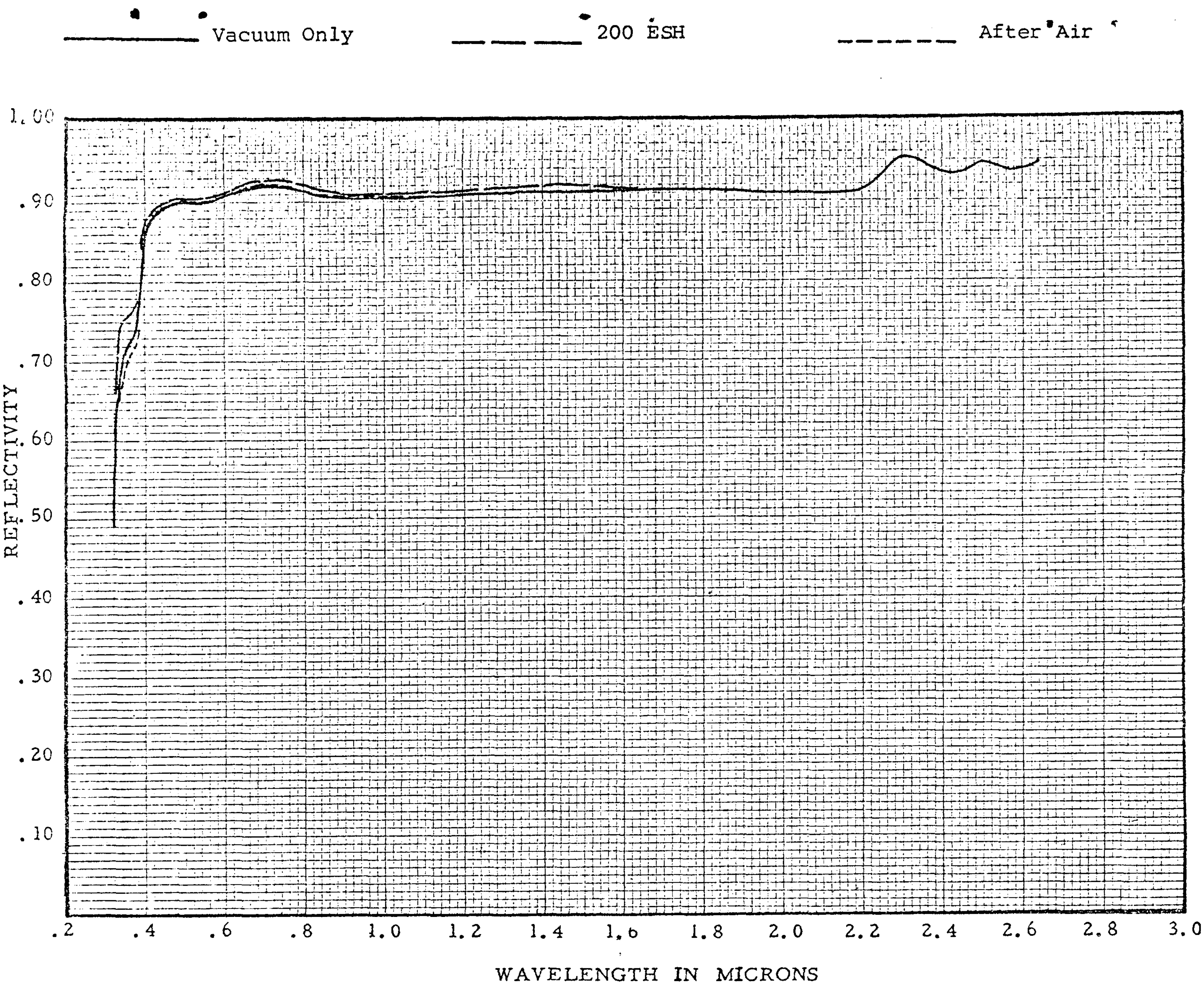


Figure 10: EFFECT OF 200 ESH OF UV IRRADIATION ON Zn_2TiO_4 POWDER SAMPLE B (IRIF TEST 2)

zinc orthotitanate. Both specimens exhibited an increase in reflectance on irradiation in the 3750-A region; the increase amounted to nearly 4% in sample B. Although this test was for only 200 ESH of ultraviolet irradiation, these results offer an excellent prognosis for continued research with the zinc titanates.

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